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Observations on the Synergistic Interactions of Aqueous Oxidizers and Ultraviolet Radiation for Decontamination Applications

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This feasibility study examines the potential benefits to result from combining aqueous oxidizers and ultraviolet radiation for decontamination applications. Thus, malathion, and organophosphorus pesticide, was exposed to systems of aqueous oxidizer with and without UV irradiation (280 — 320 nm) at a flux that approximates bright noon sunlight. Irradiated systems exhibited loss of malathion up to five (5) times as rapid as non-irradiated systems. Pseudo first-order rate constants for these systems are presented. Also, similar enhancement of substrate destruction is observed for quinine sulfate interacting with aqueous hypochlorite with and without added ultraviolet radiation. The results reported here argue strongly for the potential advantages of combining aqueous oxidizers and UV radiation for decontamination applications. Oxidizers studied were hypochlorite, perborate, peroxide, peroxydisulfate, and percarbonate anions.

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INTRODUCTION

Military operations can be compromised by contact with Chemical Warfare (CW) and/or Biological Warfare (BW) agents/toxins. Maintenance of mission effectiveness may require rapid decontamination (decon) of the personnel, equipment, and operational area. Decon becomes especially important if the extended use of protective equipment and/or enclosures hinders rapid response to changing conditions.

Decon can be passive or active. A passive decon approach allows contamination to weather away through natural processes such as atmospheric flow/wind, rainfall, solar irradiation, etc. However, time constraints usually dictate the need for more active decon measures, such as the washdown of contaminated surfaces with water, or water containing chemically active additives. Historically, the additive of choice for active decon has been hypochlorite, since aqueous hypochlorite can both oxidize and hydrolyze CW agents. Hypochlorite is also effective against BW toxins/spores (Hoffman and Spiner, 1962; Zirin, et al., 1965, Fielding, et al., 1967, 1968; Block and Davis, 1978). Thus, aqueous hypochlorite washdown exhibits a broad spectrum decon efficacy. However, the use of aqueous hypochlorite as a simple washdown may not fully utilize all the characteristics and advantages of its chemistry.

Molecules of many oxygen containing anions (e.g., peroxygen anions and hypochlorite) strongly absorb ultraviolet (UV) radiation (see Figure 1). On UV irradiation of aqueous solutions of these species, transient, energetic, and reactive intermediates such as chlorine atoms, and singlet and triplet oxygen (Ogata and Takagi, 1981) are formed. Ogata, et al. (1979), and Nakamura and Ogata (1971) have reported rapid photooxidation of certain aliphatic acids and alkylbenzene sulfonic acids to carbon dioxide and water using UV irradiation and aqueous hypochlorite. Thus, the synergistic effect of high energy, high flux UV irradiation on oxygen-containing anions such as hypochlorite could potentially be used to rapidly destroy organic compounds such as certain CW agents and BW toxins which may not directly absorb UV radiation. Note that GA (Tabun), GB (Sarin), and GD (Soman) are essentially transparent above about 220 nm, and that VX, L (Lewisite), and HD (sulfur

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mustard) are only slightly more opaque at wavelengths greater than 220 nm. The commonly used simulant dimethyl methyl phosphonate, DMMP, is also essentially transparent to UV radiation. Agent cross sections range from 10^{-21} to 10^{-17}, depending on wavelength (Rewick, et al., 1986). Therefore, one cannot expect destruction of agent by exposure to UV irradiation alone: there is insufficient sorbtion of UV radiation to cause significant destruction of chemical bonds in the agents. However, in a decon context, UV irradiation of aqueous oxidizer solutions offers both the proven chemical, and the previously unexploited photochemical, neutralization of CW/BW materials.

This report summarizes the results of a feasibility investigation to demonstrate whether decon operations could benefit from the synergistic effects of UV irradiation and an oxygen containing anionic species such as aqueous hypochlorite or peroxide. In this preliminary investigation, selected simulants for CW agents (e.g. quinine sulfate, and malathion) were reacted in aqueous oxidizer solutions with and without UV irradiation. Faster destruction of the simulants in the aqueous media containing irradiated oxidizer indicates the synergism approach to be a promising decon method. (See also Ogata and Takagi, 1981; Nadezhdin and Dunford, 1979; Mill and Gould, 1979).

EXPERIMENTAL

Two test sequences were utilized in this study. The first group of experiments used quinine sulfate as a simulant for CW agents, and the second group employed malathion. In both cases, aliquots of aqueous solutions of the simulant with and without added oxidizer were exposed to low fluxes of UV(B) radiation. This UV band (280-320 nm) is a component of solar radiation reaching the earth's surface (Figure 2), and was selected for this reason. The experimental controls were aliquots of solutions containing both simulant and oxidizer not exposed to UV(B). The disappearance of the simulant with time was monitored using fluorometry for quinine sulfate and high pressure liquid chromatography (HPLC) for malathion.

Quinine Sulfate Studies

Materials

The first group of experiments used quinine sulfate [$(C_{12}H_{22}N_2O_2)_2 \cdot H_2SO_4$] (Fisher) as the simulant, and aqueous sodium hypochlorite (NaOCl, 5% (w/v) solution, nominal, standardized by iodometric titration before use, Fisher) as the oxidizer. A stock solution of quinine sulfate (100 ppm w/w dry powder in distilled water, ~2 x 10^{-4}M) was stored in a brown glass bottle. Test aliquots were taken from this stock.

Method

Aliquots (5.0 mL each, volumetric pipette) of quinine sulfate solution were dispensed into a series of 6 cm diameter Pyrex petri dishes. This test configuration provided a fluid layer approximately 1 mm deep, and was chosen to simulate the film present when a decon fluid is applied to a surface. The quinine sulfate solutions were then divided into four groups: two groups containing only quinine sulfate solution as the control, and two containing the quinine sulfate with added hypochlorite (100 µL, Eppendorf pipette, to give 5.0 mL of solution 2 x 10^{-4}M in quinine sulfate, and approximately 1 x 10^{-2}M in hypochlorite). One set each of the control and test solutions were retained in the dark, and a similar set was exposed to a UV radiation source. The radiation source was a 48 watt General Electric fluorescent tube with a high output of UV(B)(280-320 nm). The samples were irradiated approximately 20 cm from the tube for 5 or 10 minutes. For comparison, the bright noon sun provides 1-2 Sunburn Units (SU) depending on the angle of incidence and other factors. UV(B) flux on the test samples was monitored with a Solar Light Company Model SSI 10944 light meter, filtered to respond tc UV(B). Radiation flux was measured in SU, a non-dimensional parameter. The amount of quinine sulfate in the various solutions was monitored fluorometrically as a function of time.

Measurement of Quinine Sulfate

The fluorescence from the quinine sulfate solutions was measured with an American Instruments Model J4-7439 fluorometer [excitation with a blue CS 5-60 filter (400 nm), emission was monitored through a red CS 2-64 filter (700 nm)]. The measured fluorescence from the sampled aliquot was recorded, and reported as a relative measure of quinine sulfate in solution. At the low concentration of quinine in the test solution, fluorescence is linear with concentration (Ewing, 1969).

Since the pH of 2 x 10^{-4} quinine sulfate in water is 4.8, but the pH of a test quinine sulfate plus 100 µl concentrated hypochlorite solution is ~8.6, all test reactions and control conditions were adjusted to pH ~8.6 with aqueous sodium hydroxide as needed. Furthermore, the fluorescence of quinine sulfate solutions is pH dependent, with maximal fluorescence at pH 2-3 (see Table 1). Therefore, the pH of all samples, controls, and tests was lowered to pH =3 just prior to the fluorescence measurements. All pH determinations were with a Fisher Model 355 pH meter, equipped with a Fisher combination pH electrode.

Malathion Tests

Materials

The second group of experiments utilized S-(1,2-dicarbethoxyethyl) 0,0-dimethyldithiophosphate (malathion, $C_{10}H_{19}O_6PS_2$, Foxboro Analabs) as the simulant, and various oxidizers in aqueous solution (see Table 2). The malathion test solutions were prepared by adding malathion stock solution (50 mg malathion in 10 mL acetonitrile) to 25 mL of a pH 7 phosphate buffer, giving a final malathion concentration of ~0.6 x 10^{-3} M in the test solutions. The concentrations of the oxidizers are reported in Table 2. Approximately 10 mL of these various solutions were placed in petri dishes for testing. Reaction conditions, glassware, radiation source, and procedures were essentially identical to those in the tests using quinine sulfate. Irradiated samples were covered with quartz disks to lessen evaporation of the solutions during the irradiation portion of the test sequence. Quartz is transparent to UV radiation.

Measurement of Malathion

Relative concentrations of malathion in the test solutions were monitored using high pressure liquid chromatography (HPLC). The HPLC system consisted of a Beckman Model 110A pump, a Waters Model U6K injector, a Fisher Resolvex C_{18} reverse phase column, and a Waters Model 440 UV detector filtered to 254 nm. The mobile phase for the malathion measurements was 75% methanol/25% water (v/v) and the flow rate was 1 mL/min. Prior to use, the mobile phase was degassed by stirring under vacuum. The injection sample size was 25µL (Hamilton variable syringe).

RESULTS AND DISCUSSION

Quinine Sulfate

Fluorescence data for the quinine sulfate test solutions are presented in Table 3. It is clear that with hypochlorite and UV(B) irradiation, fluorescence of the test solutions is less than those not exposed to UV(B). This difference in fluorescence is directly related to the disappearance of quinine in the irradiated hypochlorite bearing solutions. The quinine could be partially, or totally, oxidized by hypochlorite/UV(B) interaction. A partial oxidation of quinine (see Figure 3) could involve the vinyl group carbon-carbon double bond in the molecule. Such an attack could yield an alcohol, glycol, or perhaps a carboxylic acid. A more complete oxidative attack on the quinine could produce a host of molecular fragments, carbon dioxide, and related products. The limited scope of this study precluded identification of such reaction products.

Malathion

Malathion, as in the case of quinine, underwent an accelerated attack with oxidizer plus UV irradiation for all oxidizers except sodium percarbonate (see Figure 5-a). The relevant pseudo-first order rate constants for these interactions are listed in Table 4, and one sees oxidation rates two to five times faster with UV irradiation of the oxidizer system than without (see section on "Analysis of Data for Malathion," for detail). This rate enhancement, it must be emphasized, is at very low UV flux, a flux which roughly corresponds to the amount of UV radiation available on a bright, sunny day. With higher UV flux, the monitored reaction may proceed much more rapidly. This possibility should be examined in more detail. Note, however, that malathion reacted rapidly with sodium percarbonate with or without added UV irradiation.

The rate constants in Table 4 indicate that, to a first approximation, malathion reacted with hydrogen peroxide and sodium perborate at the same rate, with or without UV irradiation. Similarly, the reactions of malathion with sodium peroxydisulfate and with sodium percarbonate in the presence of UV irradiation exhibited comparable, but faster reaction rate constants. The observed enhancement with these latter species may be due to a different molecularcoupling mechanism by which the UV radiation is transferred to the malathion substrate. It would be useful to examine in more detail the actual mechanism by which the peroxide oxidizers interact destructively with the UV radiation, and the substrate.

It is postulated that the destruction of malathion could involve conversion to malaoxon (R. Landolt, pers. comm., 1985), with the double bonded sulfur to phosphorous being replaced by a double bonded oxygen to phosphorous (see Figure 3). If the oxidative attack on malathion stopped at malaoxon, it should be possible to detect this product by HPLC. However, when malathion interacted with hypochlorite, no peaks other than hypochlorite and malathion were ever recorded on the chromatograms from the pre-and post-irradiation solutions. Further, note from Figure 4 that the hypochlorite signals in the two traces have not changed appreciably in size or shape. This fact suggests that the hypochlorite might be acting catalytically by transferring UV energy to the malathion, but not itself being consumed. If the UV-hypochlorite synergism is catalytic, then a decon utilizing this approach would prove effective with no or minimum consumption of hypochlorite.

CONCLUSIONS

The experiments described in this feasiblity study indicate aqueous hypochlorite and UV(B) radiation appear to interact synergistically in the destruction of certain organic compounds. Neither aqueous quinine sulfate nor malathion was consumed by exposure to either UV(B) radiation or aqueous oxidizer alone. However, both test compounds were decomposed rapidly when exposed to the synergistic effects of UV(B) irradiation of the oxidizer solution.

RECOMMENDATIONS

The concept of synergistic oxidative attack on organic compounds by UV radiation combined with aqueous oxidizer needs to be further developed for decon applications. Required are studies which:

1) Examine the observed synergism between UV and the aqueous oxidizers under optimal conditions. UV flux, wavelength, and band pass, as well as oxidizer concentration will affect overall efficacy of UV/oxidizer decon, and must be investigated. A most efficient oxidizer for use with UV irradiation needs to be identified.

2) Extend the observations concerning synergistic destructive attack on aqueous quinine sulfate and malathion to other challenges of interest. Appropriate simulants for CW agents such as 2-chloroethyl ethyl sulfide (CEES, a mustard simulant), dimethyl methyl phosphonate (DMMP, a G-agent simulant), and diisopropyl fluorophosphate (DFP, also a G-agent simulant), and simulants for BW toxins/spores are of vital importance and should be examined. Ultimately, the observations reported here, and to be obtained in further studies, must be confirmed with live agents and biological toxins.

3) Investigate the possibility that synergistic UV/oxidizer decon may be catalytic. If the process is catalytic, then small amounts of oxidizer could serve to decon large surface areas, with decon being limited only by the amount of UV irradiation applied to the system.

Further phases of the project could include, but not be limited to, the design of appropriate ultraviolet radiation sources for use with hypochlorite decon. Such sources could range from hand-held, battery operated units for effective spot-decon, as at passageways or cockpits, to larger banks of UV sources, allowing the rapid, more effective decon of exterior surfaces such as bulkheads, work stations, or supply storage areas. The availability of commercial UV units should be explored and selected units tested.

ANALYSIS OF DATA FOR MALATHION

The pseudo-first order rate constants reported in Table 4, and the graphical results presented in Figures 5-9 were generated in the following manner, using data from Table 5. Let the initial concentration of malathion be c_0 in any test system. At any time "t" after the start of a particular test, one can determine a malathion concentration in the test solution; this concentration is c. In some cases, $c \cong c_0$, but in general $c < c_0$, especially for the irradiated systems. If one plots $\ln c/c_0$ vs. t, the slope, k, of this line is the rate constant for the reaction. Therefore, $k = \ln(c/c_0)/t$, with k in units of reciprocal time, $days^{-1}$, in this case.

Table 1 - Quinine* Fluorescence as a Function of pH

pH	Fluorescence
2.20	16.6
2.27	16.5
2.45	16.0
2.71	15.5
3.07	14.6
4.80	0.35
5.00	0.22
5.57	0.08
6.50	0.004
6.73	0.003
7.07	0.002
7.36	0.001
7.59	~0
8.04	~0
8.44	~0

*Quinine sulfate is 100 ppm or 1 x 10^{-2}M. For quinine, pK_1 = 8.52; pK_2 = 4.13

Table 2 - Oxidizers Used in This Study

Oxidizer, formula, source	F.W.	Concentration used in test, N
Sodium Hypochlorite, NaOCl Fisher (5% solution)	74.5	0.01
Hydrogen Peroxide, H_2O_2 Fisher (30% solution)	34.0	0.102
Sodium Peroxydisulfate, $Na_2S_2O_8$ Alfa	238.0	0.076
Sodium Perborate, $NaBO_3 \cdot 4H_2O$ Alfa	153.8	0.047
Sodium Percarbonate, $2Na_2CO_3 \cdot 3H_2O_2$ Burlington	314.0	0.083

Sources:
Fisher Scientific Company, Fairlawn, NJ
Alfa Products, Danvers, MA
Burlington Chemical Company, Burlington, NH

Table 3 - Synergistic Effect of UV and HOCl on Quinine Sulfate Fluorescence Data for Quinine Sulfate Tests

	5 min.	10 min.
no UV-B	1.3* ± 0.2	1.75 ± 0.2
with UV-B, 1 SU	0.43 ± 0.07	0.45 ± 0.06

*All fluorescence data are averages of three separate runs.

Table 4 - Pseudo First-Order Rate Constants for Interaction of Malathion and Selected Oxidizers

System	k, in days^{-1}
A. Malathion only	0.027
Malathion + UV	0.013
B. Malathion + hydrogen peroxide	0.61
Malathion + peroxide + UV	1.64
C. Malathion + peroxydisulfate	0.61
Malathion + peroxydisulfate + UV	5.0
D. Malathion + perborate	0.66
Malathion + perborate + UV	1.0
E. Malathion + percarbonate	--
Malathion + percarbonate + UV	4.72

Table 5 - Data for Malathion/Oxidizer/UV Synergism Studies

Time (days)	Concentration*	Time (days)	Concentration*
A. Malathion only		+ UV irradiation	
0	600	0	605
1.0	620	1.0	610
1.9	630	2.1	620
2.9	610	3.0	590
4.2	630	4.2	590
6.0	590	6.0	580
B. Malathion + hydrogen peroxide		+ UV irradiation	
0	570	0	570
0.9	430	1.0	100
1.9	270	2.1	n.d.
2.9	130		
4.1	50		
5.9	n.d.*		
C. Malathion + peroxydisulfate		+ UV irradiation	
0	490	0	490
0.9	300	0.9	4
1.8	190	2.0	n.d.
2.8	120		
4.1	50		
5.9	10		
D. Malathion + perborate		+ UV irradiation	
0	560	0	560
0.9	460	0.9	220
1.9	260	2.0	n.d.
2.8	130		
4.0	40		
5.9	n.d.		
E. Malathion + percarbonate		+ UV irradiation	
0	530	0	530
0.8	n.d.	0.9	10
1.9		1.9	n.d.

*Concentration is the number of micromoles contained in a 25 µL injection of test solution withdrawn from the bulk sample (10 mL) held in petri dishes either with or without UV irradiation.
n.d. = not detected.

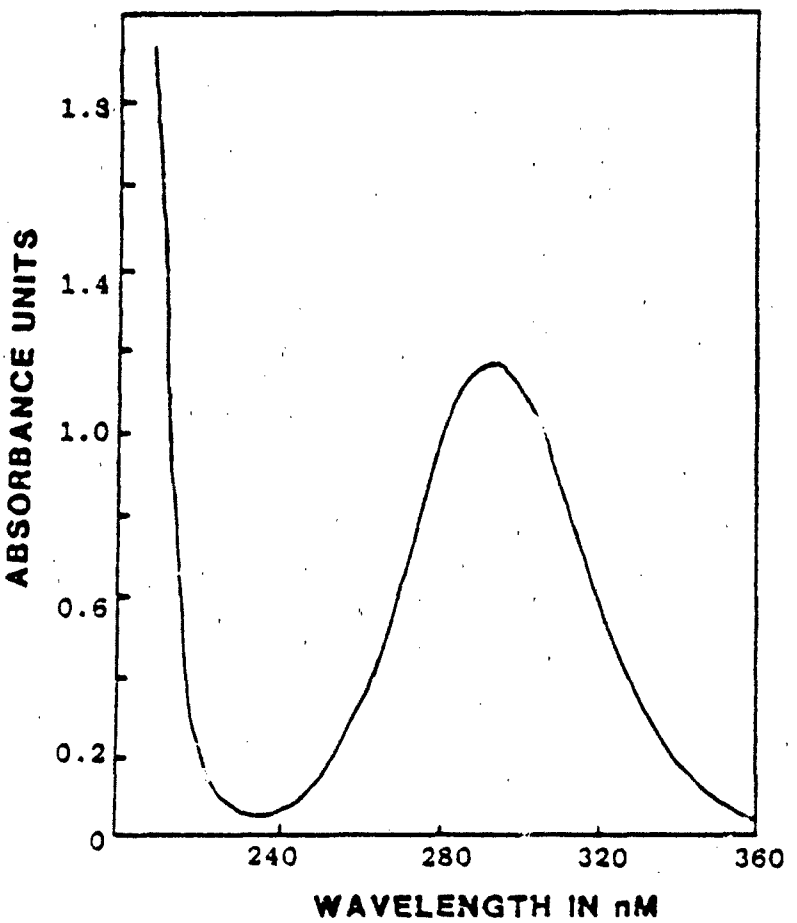


Fig. 1 — UV absorption spectrum of aqueous hypochlorite ion

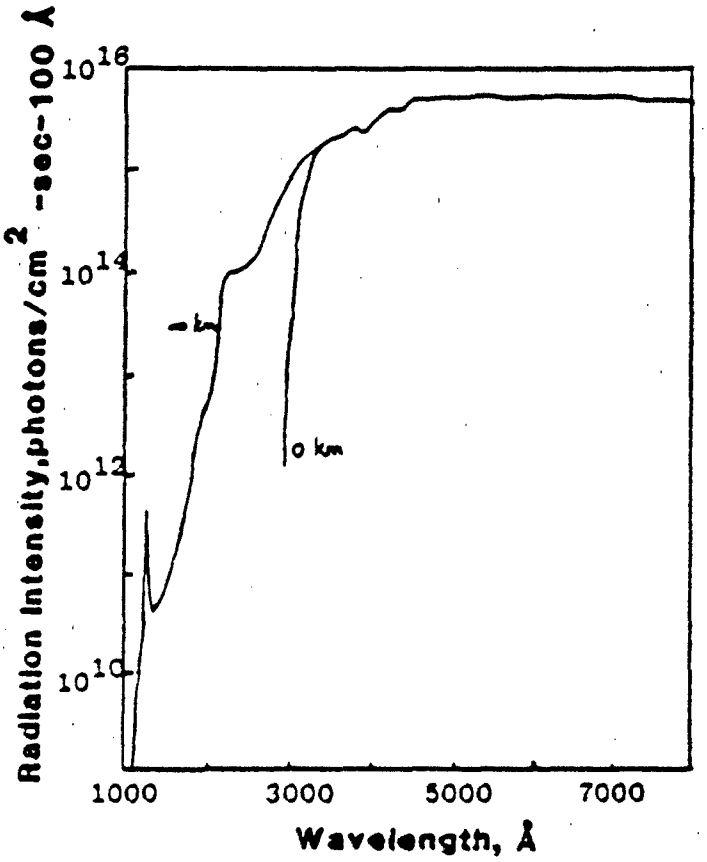
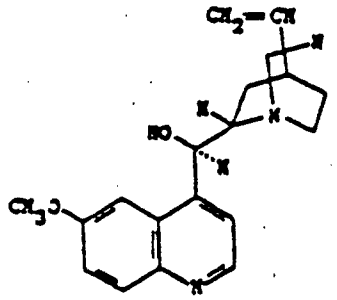
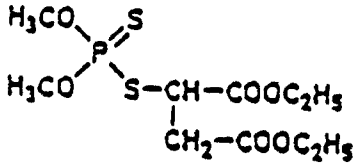


Fig. 2 — Relative intensity of solar radiation as a function of wavelength, at earth's surface (0km) and upper atmosphere (∞km)

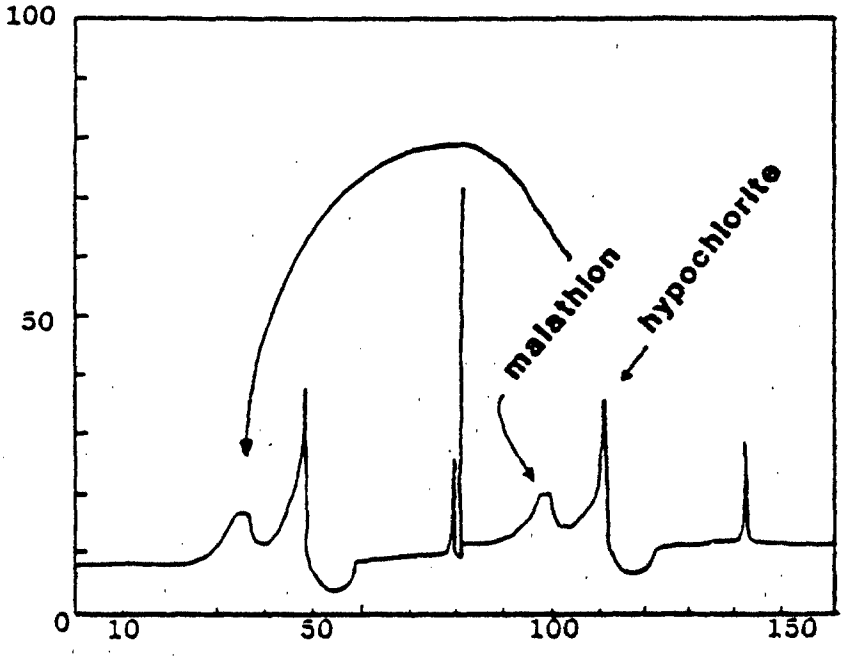


QUININE

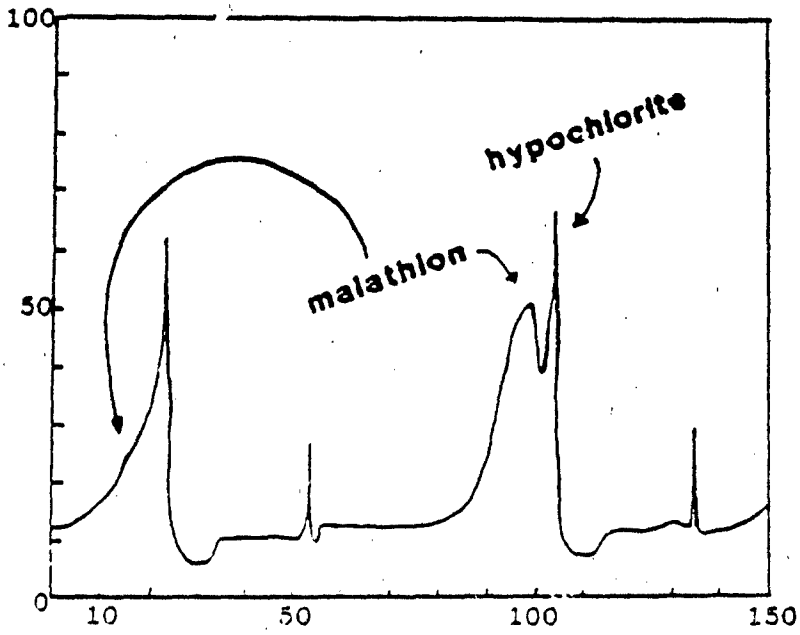


MALATHION

Fig. 3 — Molecular structures of test compounds



a. Malathion and hypochlorite without UV irradiation. Approximately 45 minutes between injections of samples stored in the dark. Earlier assays are on the right of the chart.



b. Malathion and hypochlorite system irradiated by UV. Note decreases in malathion signal after only 5 minutes irradiation.

Fig. 4 — HPLC chromatograms of malathion/hypochlorite/UV irradiation systems

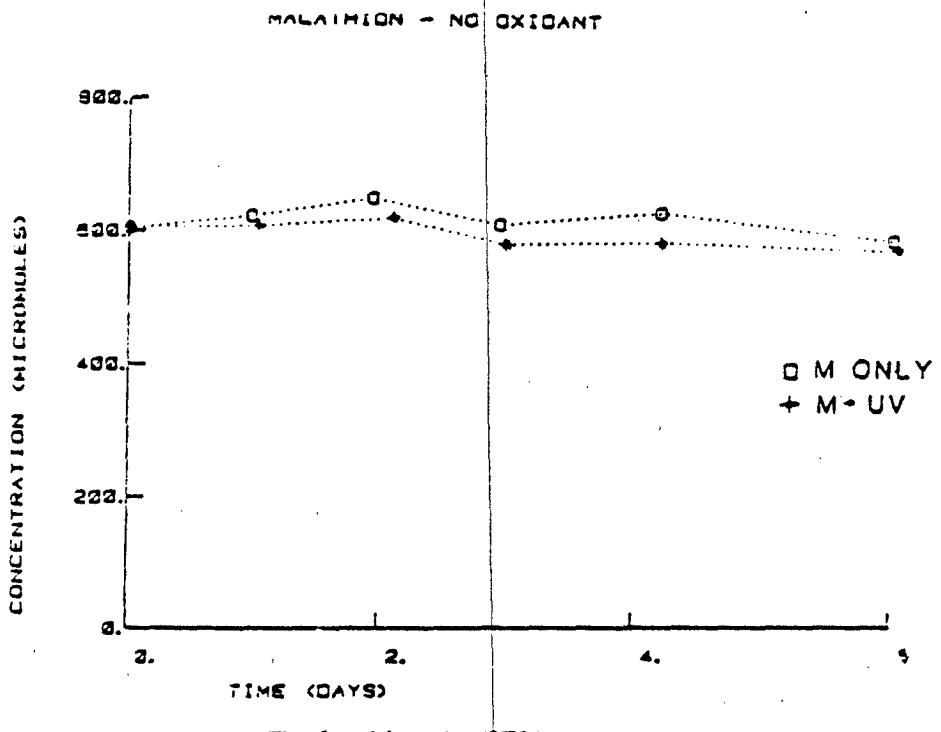


Fig. 5 — Malathion/UV interactions

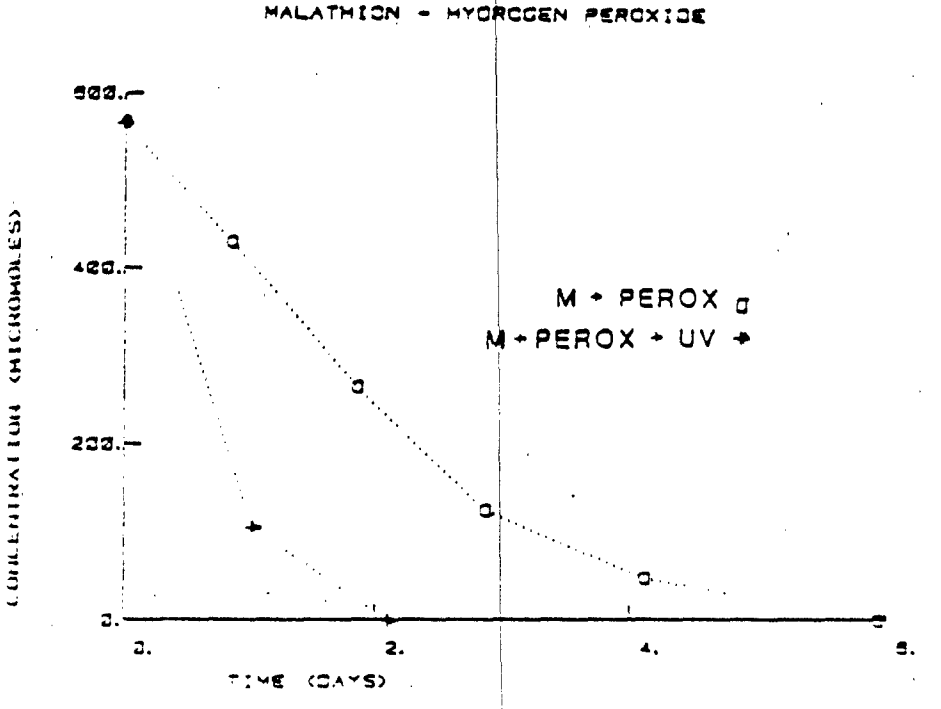


Fig. 6 — Malathion/hydrogen peroxide interactions

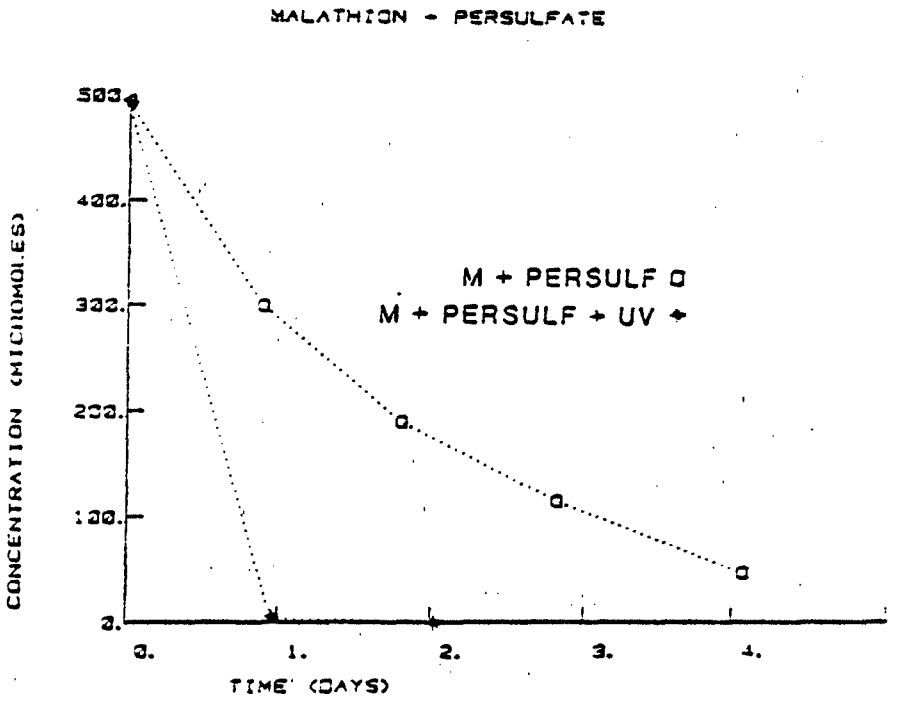


Fig. 7 — Malathion/sodium peroxydisulfate interactions

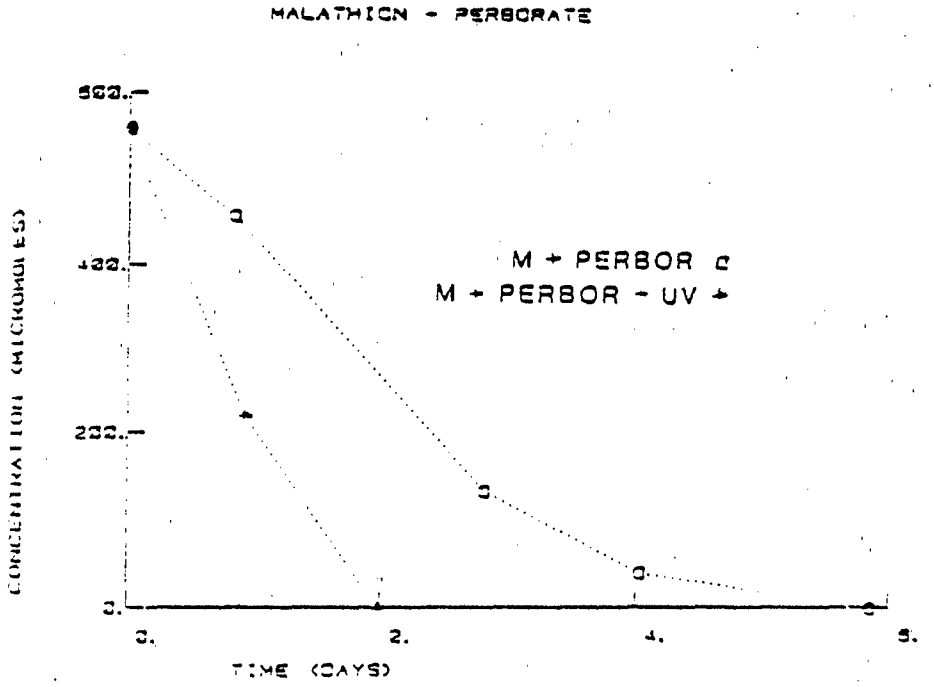


Fig. 8 — Malathion/sodium perborate interactions

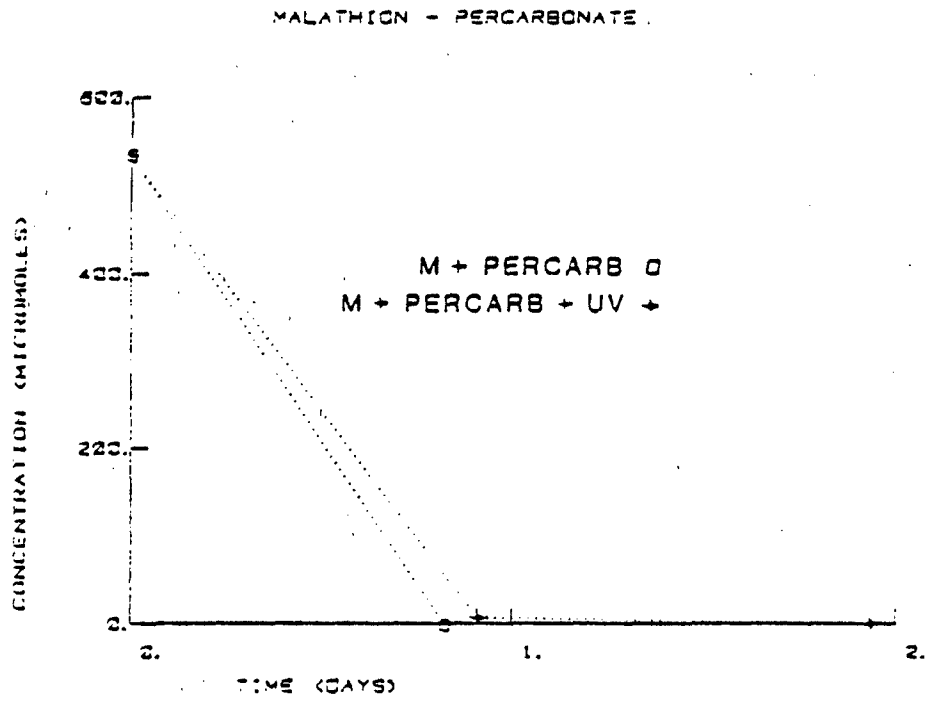


Fig. 9 — Malathion/sodium percarbonate interactions

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